

Concentrations of PCDD and PCDF
in Soil from the Vicinity of the
SWARU Incinerator, Stoney Creek,

September, 1985

Report No. ARB-013-85-Phyto



Ministry
of the
Environment

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*Concentrations of PCDD and PCDF in Soil from the Vicinity
of the SWARU Incinerator, Stoney Creek*

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Date: September, 1985

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of the SWARU Incinerator, Stoney Creek***

BACKGROUND INFORMATION

In the spring of 1983 the Phytotoxicology Section was requested to conduct an assessment survey in the vicinity of the SWARU refuse incinerator in Stoney Creek, to determine if polychlorinated dibenzo-p-dioxins (PCDDs) or polychlorinated dibenzofurans (PCDFs) were present in surface soils. The incinerator began operation in 1973.

The collection of soil for high resolution analysis of complex organic compounds such as PCDDs and PCDFs requires an extremely elaborate sampling protocol. Although the Phytotoxicology Section has had extensive experience in assessing soil-borne contaminants, terrestrial samples for PCDD/PCDF analysis had never previously been collected in Ontario. However, PCDD contamination has occurred in the United States and the EPA has developed a protocol to meet their soil sampling requirements. Therefore the officials involved with the EPA's dioxin sampling program were consulted by Phytotoxicology investigators and the resultant information exchange enabled the MOE to develop a sampling protocol suitable to Ontario's environmental conditions.

SAMPLING PROCEDURE AND PRECAUTIONS

Briefly, the sampling procedure for PCDDs and PCDFs differed from that used for other soil surveys in that the sampling equipment was scrubbed laboratory-clean between each site to ensure that no cross-sample contamination occurred. This was accomplished by washing the sampling equipment with an alconox solution and rinsing it with distilled water. The washing step was repeated until all visible soil residue was removed. This was followed by a rinse with 95% denatured alcohol to completely dry the equipment. To remove any residual trace organics which may have adhered to the chromed surface of the sampling corer it was then rinsed with 1,1,1-trichloroethane.

To further minimize any possibility of cross-site contamination all sample locations were assumed to be contaminated relative to their distance from SWARU; accordingly within the survey area the sites farthest from SWARU were sampled first, followed by progressively closer locations. The field personnel wore clean disposable plastic gloves at each site so that the soil was never touched by unprotected hands.

A 2 m diameter circular plot was established at each of the 14 sample locations. Actual sample collection from within each plot consisted of 15 to 20 soil plugs 2 cm in diameter and 5 cm in depth, yielding approximately 500 g of soil. The soil was placed in a wide-mouth, amber-coloured glass jar which had been solvent-rinsed in preparation for organic samples. The sample jar lid was foil-lined and secured with tape when sample collection was completed. All filled sample jars were placed in separate plastic bags and stored in an insulated, light-tight cooler until delivery to the MOE dioxin laboratory.

ANALYTICAL PROTOCOL

The MOE Dioxin Facility does not have an operational protocol to determine PCDD and PCDF in soil. Therefore the soil samples collected

from around SWARU were analyzed by a private laboratory, Zenon Environmental. This laboratory was tested with spiked check soil samples and their performance was found to be satisfactory before they began work on the SWARU soil samples.

The following is a brief summary of the analytical procedure used by Zenon Environmental as provided by Dr. R.E. Clement, MOE Dioxin Laboratory. A description of the entire analytical procedure is available in the appendix to this report.

The samples were received, ground, weighed, air dried and numbered at the MOE Dioxin lab and shipped to Zenon. At Zenon each sample was Soxhlet extracted using toluene. This was followed by a four step multiple column chromatographic clean-up. Some soil extracts required additional treatment with activated copper and AgNO_3 to remove sulfur. Quantification to the low parts per trillion range was made with gas chromatography/mass spectrometry.

Isomer specific identification was not attempted, rather total concentrations of five congener groups each of PCDDs and PCDFs were obtained. The congener groups included tetraCDD (T_4CDD), pentaCDD (P_5CDD), hexaCDD (H_6CDD), heptaCDD (H_7CDD) and octaCDD (O_8CDD). Similarly the PCDF congener groups were tetraCDF (T_4CDF), pentaCDF (P_5CDF), hexaCDF (H_6CDF), heptaCDF (H_7CDF) and octaCDF (O_8CDF).

SITE SELECTION

The MOE Hamilton Office requested that some soil samples be collected from the areas where residents have complained about fly ash fallout. Samples also were collected at various distances from SWARU to determine if a soil contamination gradient was present. All sample sites were fully exposed to aerial deposition and were either sodded or had natural grass and sedge cover. No private residential properties were sampled.

Fourteen soil sites were chosen and the sampling was conducted on July 7 and 8, 1983. A description of each site is listed in Table 1 and the locations are illustrated in the attached map.

Two of the 14 sites were urban control locations in Stoney Creek. These were located 3 to 4 km beyond the calculated maximum ground level concentration of emissions from the SWARU incinerator. One additional site was a remote, rural control location selected to reflect the levels of PCDDs and PCDFs in soil not exposed to urban activity.

In addition to the samples of soil, a sample consisting of ash residues from the SWARU plant which were stockpiled on company property to the E of the stack was collected for analysis.

RESULTS OF ANALYSIS

The results of the soil analyses are summarized in Table 2. The data are in parts per billion (dry weight basis) for each of the five PCDD and PCDF congener groups. The MOE does not have a guideline or any other criterion/objective for PCDDs or PCDFs in soil. The U.S. EPA have used the level of 1.0 ppb for 2,3,7,8-TCDD. This was developed by the Atlanta Center for Disease Control as the level for concern regarding human exposure to the most toxic of the 22 possible tetraCDD isomers. No guidelines of any kind exist for any of the other PCDD or PCDF isomers or congener groups.

All 14 of the soil samples had detectable quantities of at least one of the five PCDD congener groups whereas eight samples contained detectable levels of one or more PCDF congener groups. Only one site (No. 5 -1570 m SSE) had a measurable quantity of tetraCDD in the soil. The concentration at that site was 0.007 ppb T₄CDD.

The highest PCDD concentration was 3.5 ppb octaCDD detected at Site 11, 1260 m SW of SWARU. However, a similar level of 3.2

ppb octa CDD was found in the soil at one of the urban control sites (No. 8, 5570 m ESE) well remote from the SWARU plant. The soil at the remote/rural control site which was located at the bottom of a 50 m ravine in Bronte Creek Provincial Park, approximately 22 km N of SWARU, contained 0.810 ppb octaCDD. The levels of the other PCDD and PCDF congener groups in soil from the 11 sites around SWARU generally were within the range detected in the urban control soils. There were no apparent concentration gradients for any of the PCDDs or PCDFs when compared with distance or direction relative to SWARU or when considered in terms of the prevailing wind directions (see attached map).

The location of the maximum ground level concentration (c-max) for emissions from the SWARU stack has been calculated* at 1182 m. The c-max is illustrated on the attached map as a circle around SWARU. The five sample sites closest to the c-max are No's. 2,5,6,10 and 11. Only two of these sites (No's. 5 and 11) had detectable levels of more than one PCDD congener group. Neither of these two sites was located in the direction of the prevailing winds, which for the last nine years have blown towards the E, NE and N (in the general direction of Lake Ontario) over 50% of the time. In addition, soil at Site No. 2 which is only 160 m from No. 11, contained less than one tenth the octa CDD concentration and no other PCDDs or PCDFs.

The single sample of incinerator ash residue (see appendix - sample No. 2351) collected on SWARU property had total PCDD and PCDF concentrations of 2.8 and 2.9 ppb respectively, for a PCDF to PCDD ratio of 1.04. Ozvacic *et al.* (1984) determined that the levels of PCDFs in SWARU precipitator fly ash and stack particulate emissions were about twice the concentration of PCDDs, for a PCDF to PCDD ratio approximating 2.0. Assuming that the environmental fate of these compounds is similar (no information exists in the literature to confirm or refute this assumption) then soil contaminated with PCDDs from SWARU emissions should contain similar if not greater concentrations of PCDFs. As none of the soil samples had a PCDF to PCDD ratio higher than 0.39 and as five of the eleven sites

* Environmental Protection Act, 1971: Reg. 308

(excluding control locations) contained no measurable PCDFs there does not appear to be any relationship between the PCDDs and PCDFs found in the soil and those known to be emitted from SWARU. The data appear to reflect the general ubiquity of these compounds in urban soil.

LITERATURE RETROSPECT

To date only a limited amount of information has been published concerning the fate and persistence of PCDDs in soils. The information which is available is further limited to the behavior of only one of the 75 possible PCDD isomers, the most toxic 2,3,7,8-TCDD. The following points briefly summarize the behavior of this chemical in soils (Kimbrough *et al.*, 1984; Young, 1983; Young and Arnold, 1983; Wipf *et al.*, 1982):

1. it has been shown to be extremely persistent in soils with an estimated half-life of over 10 years; however, the results of recent studies suggest that even this may be an underestimate of half-life as new, more vigorous extraction techniques have now recovered TCDD originally thought to have been lost from soils.
2. it is fairly rapidly photo-degraded on exposure to UV light provided it is solubilized with an effective hydrogen donor.
3. it is metabolized in soil to only a very limited degree by a few strains of soil microbes.
4. its vertical movement in soil is fairly limited due to its strong adsorption to soil particles; however it has been found to migrate in sands to a depth of 30 cm presumably due to physical transport.
5. it appears to be only slightly accumulated by root plants with most of the contamination being detected in the peel of soil-exposed roots and tubers.

Soil contamination by PCDD has been documented in the vicinity of chlorophenol production industries, at military test sites following massive applications of phenoxyacetic acid herbicides, and as a result of spills and improper disposal of contaminated waste oils. Most of the published data refer specifically to 2,3,7,8-TCDD. Table 3 lists some of the available data and compares the findings with soil levels around the SWARU plant. The first four lines of Table 3 represent a summary of soil concentrations found around a chlorophenol manufacturer, agent orange herbicide test spray and storage areas and in the vicinity of the Seveso accident. The data reveal that levels may range up to several thousands times higher than those detected in Stoney Creek. The fifth line of Table 3 is soil data in the vicinity of incinerators in Chicago. The highest Stoney Creek soil PCDD levels compare with the lower end of the range of the Chicago data. There are virtually no published data on PCDF soil concentrations.

CONCLUSIONS

This was the first soil survey for PCDD and PCDF conducted by the Ministry of the Environment. A sampling protocol had to be developed and the collected soil samples were analyzed at a private laboratory (Zenon Environmental).

PCDDs were found in soil at all 14 sites, including three control locations. PCDFs were detected at eight of the 14 sites, including both of the urban control locations but not at the remote/rural site. The most frequently occurring congener groups were hepta and octaCDD. Only one site contained detectable quantities of tetraCDD with a concentration of 0.007 ppb, a level well below the U.S. EPA guideline of 1.0 ppb for the most toxic TCDD isomer 2,3,7,8-TCDD.

On the basis of the PCDD and PCDF analyses results for the 11 survey area and 3 control site soil collections and the absence of any concentration gradient or deposition pattern consistent with either prevailing winds or the location of the calculated maximum ground level concentration it is concluded that emissions from the operation of the SWARU incinerator since 1973 have not accumulated in surface soils in the vicinity of the plant.

REFERENCES CITED

Dow Chemical. 1978. *The Trace Chemistries of Fire - A Source of and Routes for the Entry of Chlorinated Dioxins into the Environment.* pp 46.

Kimbrough, R.D., Falk, H., Stehr, P., and G. Fries. 1984. Evaluating the Human Health Risk of Varying Concentrations of 2,3,7,8-tetrachlorodibenzodioxin (TCDD) Levels in Soil. Submitted to *Journal of Toxicology and Environmental Health*.

Ozvacic, V., Wong, G., Tosine, H., Clement, R., Osborne, J., and S. Thorndyke. 1984. Determination of Chlorinated Dibenzo-p-Dioxins, Chlorinated Dibenzofurans, Chlorinated Biphenyls, Chlorobenzenes and Chlorophenols in Air Emissions and Other Process Streams at SWARU in Hamilton. Joint Report by Air Resources and Laboratory Services and Applied Research Branches, M.O.E. and Ontario Research Foundation. Report No. ARB-02-84-ETRD.

Wipf, H.K., Homberger, E., Neuner, N., Ranalder, U.B. Vetter, W. and J.P. Vuilleumier. 1982. TCDD-Levels in Soil and Plant Samples from the Seveso Area in Chlorinated Dioxins and Related Compounds - Impact on the Environment eds. O. Hutzinger, R.W. Frei, E. Merian, F. Porchiari. Pergamon Press, Oxford. pp 115-126.

Young, A.L. 1983. Long-Term Studies on the Persistence and Movement of TCDD in a Natural Ecosystem in Human and Environmental Risks of Chlorinated Dioxins and Related Compounds. eds. R.E. Tucker, A.L. Young, A.P. Gray. Plenum Press, New York. pp 173-190.

Young, A.L. and E.L. Arnold. 1983. *Environmental Fate of TCDD - Conclusion from Three Long Term Field Studies. Preprint Extended Abst. American Chemical Society, Washington, D.C. pp. 87-90.*

DLMcL/hm

Attach.

PH/45/1

TABLE 1 - LOCATION AND DESCRIPTION OF SOIL SAMPLE SITES

Site No.	Distance & Direction from SWARU	Site Description
1.	2100 m SW	- in valley bottom off Potruff Rd., opposite Eugene Ct., 26 m along foot path from end of truck trail.
2.	1100 m SW	- Melvin Ave. and Potruff Rd., 50 m S of Melvin opposite centre hydro pole (parkland).
3.	2480 m SW	- Queenston Rd. and Reid Ave., 50 m S of Queenston opposite centre hydro pole (parkland).
4.	2020 m SSE	- Battlefield Creek Park, 8 m W of centre pole (of three) adjacent to creek.
5.	1570 m SSE	- municipal parkette on Lake Ave., 25 m from edge of road opposite 187 Lake Ave.
6.	880 m SE	- 28 m S of first hydro tower on hydro R.O.W. S of Cascade St.
7.	2380 m E	- 9 m N of sidewalk in centre of industrial lawn at 191 Barton St. E.
8.	5570 m ESE	- urban control , Fruitland Public School, 12 m E of parking lot (school now demolished).
9.	2140 m NE	- 27 m W of access to N Service Rd off of Grays Rd.
10.	1260 m NE	- Confederation Park, 33 m E of concrete block building by main parking lot.
11.	1260 m SW	- Hillcrest Park, 30 m E of gate separating Hillsdale School and Roxborough Park Presbyterian Church.
12.	4450 m SW	- urban control , Cunningham Public School, 16 m E of gate in NW corner of school yard.
13.	70 m W	- Kenora Ave. entrance to SWARU, 9 m from edge of road (lawn area).
14.	22,000 m N	- rural/remote control valley bottom of Bronte Creek, Bronte Creek Prov. Park, approx. 1.0 km N of QEW highway.

- all sites were sodded or naturally grass covered.

- soil (0-5 cm depth) collected from within a 2 m diameter sample area.

TABLE 2 - POLYCHLORINATED DIBENZODIOXINS AND FURANS IN SOIL* AROUND
THE SWARU INCINERATOR IN STONEY CREEK, JULY, 1983

Site No.	Distance & Direction from SWARU	Site Description	Soil Concentration: ppb**											
			tetra	penta	PCDD hexa	hepta	octa	Total PCDD's	tetra	penta	PCDF hexa	hepta	octa	Total PCDF's
13	70 m W	- industrial lawn	nd	nd	nd	0.096	0.110	0.206	0.071	nd	nd	nd	0.009	0.080
6	880 m SE	- hydro R.O.W.	nd	nd	nd	nd	0.120	0.120	0.043	nd	nd	nd	nd	0.043
2	1100 m SW	- municipal park	nd	nd	nd	nd	0.310	0.310	nd	nd	nd	nd	nd	nd
10	1260 m NE	- municipal park	nd	nd	nd	0.150	nd	0.150	nd	nd	nd	nd	nd	nd
11	1260 m SW	- municipal park	nd	0.580	0.170	0.390	3.50	4.64	nd	nd	nd	0.180	nd	0.180
5	1570 m SSE	- municipal park	0.007	nd	nd	0.042	0.140	0.189	nd	nd	nd	nd	nd	nd
4	2020 m SSE	- municipal park	nd	nd	nd	0.042	1.30	1.342	nd	nd	nd	nd	0.005	0.005
1	2100 m SW	- undisturbed greenbelt	nd	nd	nd	nd	0.075	0.075	nd	nd	nd	nd	nd	nd
9	2140 m NE	- highway R.O.W.	nd	nd	nd	nd	0.050	0.050	nd	nd	nd	nd	nd	nd
7	2380 m E	- industrial lawn	nd	nd	nd	nd	1.00	1.00	nd	nd	nd	nd	0.033	0.033
3	2480 m SW	- municipal park	nd	nd	nd	0.270	0.690	0.96	nd	nd	nd	0.150	nd	0.150
Urban Control														
12	4450 m SW	- school yard	nd	nd	nd	0.005	0.940	0.945	0.009	0.006	nd	nd	nd	0.015
8	5570 m ESE	- school yard	nd	nd	nd	0.097	3.20	3.297	0.068	nd	nd	nd	0.081	0.149
Remote (Rural) Control														
14	22,000 m N	- undisturbed greenbelt	nd	nd	nd	nd	0.810	0.810	nd	nd	nd	nd	nd	nd
Analytical Detection Limit: ppb			0.0003	0.0013	0.0013	0.0013	0.0013		0.0003	0.0013	0.0013	0.0013	0.0008	

* surface soil (sodded), 0 to 5 cm depth

** parts per billion, dry weight

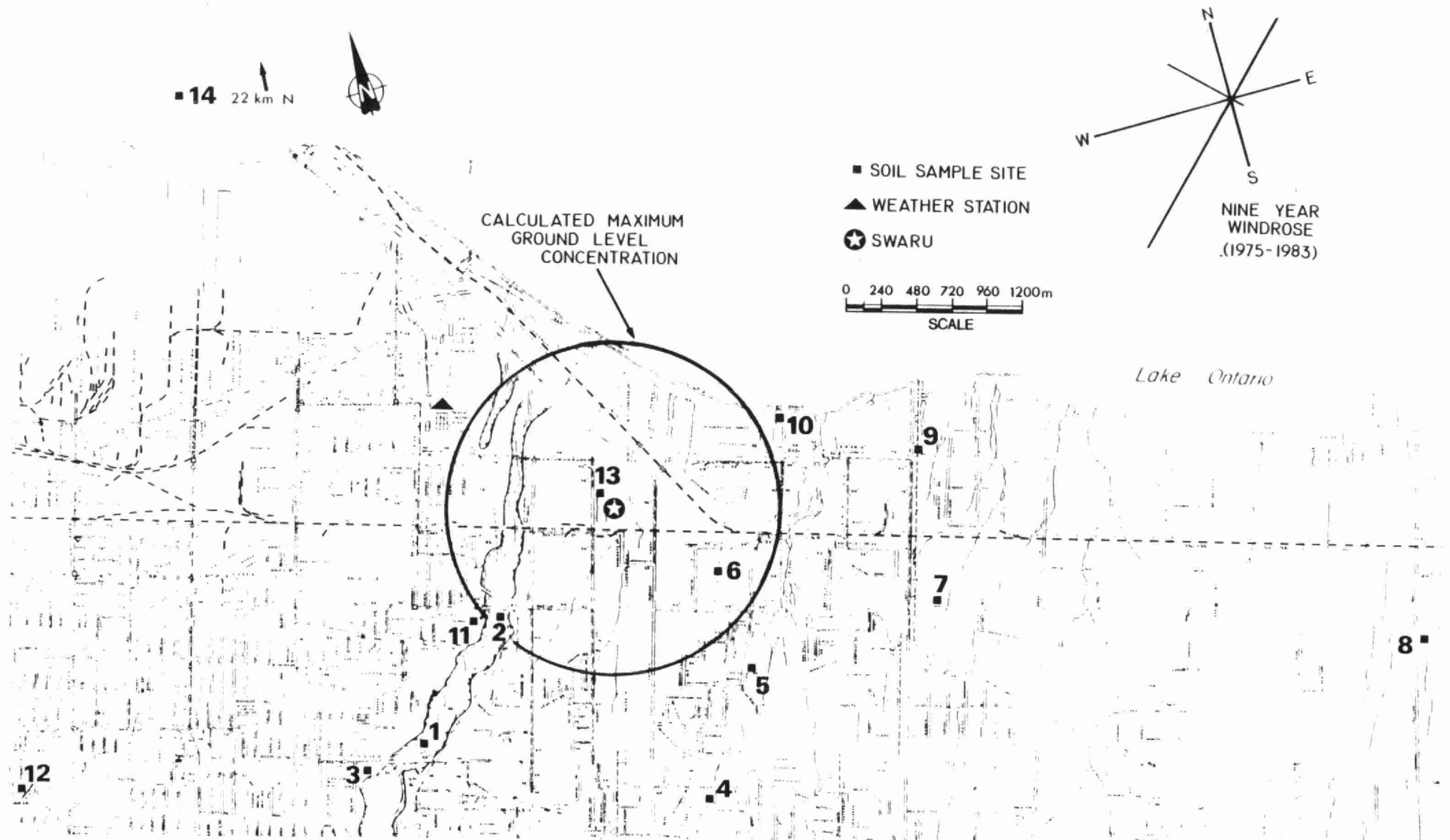
nd not detected, less than the analytical detection limit

Site No., refer to map

TABLE 3 - COMPARISON OF PCDD CONCENTRATIONS IN SOIL FROM PUBLISHED LITERATURE

Source	PCDD Concentrations in Soil (ppb)				Reference
	T ₄ CDD	H ₆ CDD	H ₇ CDD	O ₈ CDD	
<u>Midland, Michigan</u> chlorophenol manufac. industry (0-1 cm)	1.1-118	7-280	70-3200	490-20,500	Dow Chem. (1978)
<u>Eglin Air Force Base, Florida</u> Agent Orange herbicide - test spray site (Grids I, II, IV) (0-15 cm)	0.01-1.5 0.01-.47 0.01-.15				Young (1983)
<u>Gulfport, Mississippi</u> herbicide spill at storage facility (0-8 cm)	240 (Ave.)				Young <u>et al.</u> (1983)
<u>Seveso, Italy</u> Industrial accident (highest level - Zone A) (0-7 cm)	55				Wipf <u>et al.</u> (1982)
<u>Chicago, Illinois</u> Incinerators (0-1 cm)	0.005-0.03	0.03-0.31	0.14-3.3	0.35-22	Dow Chem. (1978)
SWARU survey (highest levels) (0-5 cm)	0.007	0.170	0.390	3.50	

LOCATION OF SOIL SAMPLE SITES



APPENDIX I

ANALYTICAL METHOD FOR SWARU SOIL SAMPLES

Sample Storage and Pre-Treatment

Soil samples were collected on July 7 and 8, 1983 in pre-cleaned amber sediment jars with foil-lined screw-cap tops (standard MOE stores issue). After collection, jars were kept in an insulated box for transport to the laboratory. Samples as received in jars were then stored in a freezer at approximately -10°C for about five months until analyzed.

An external laboratory was chosen to perform the extraction and GC-MS analysis of the soil samples based upon cleanup and analysis of 10g soil samples spiked by MOE with ^{13}C -2,3,7,8-TCDD and ^{13}C -OCDD. Two laboratories were initially contacted for this test but after delivery of the test samples, one of these could not perform the work due to a lack of proper standards. The other laboratory, Zenon Environmental, was chosen to analyze the remaining samples based on satisfactory performance on the check samples. Average recoveries of the MOE spiked compounds for check samples were $46 \pm 9\%$ (^{13}C -2,3,7,8-TCDD) and $66 \pm 20\%$ (^{13}C -OCDD).

Before samples were delivered to Zenon Environmental, they were manually ground using mortar and pestle. Most samples contained a small percentage of vegetation (grass) which was ground together with the particulate material. The grinding was performed until particles passed

through a standard sieve (mesh size 0.08 inch). Between samples, the mortar and pestle and sieve were rinsed with water followed by methanol. After rinsing, a few grams of the next sample were ground, passed through the screen, then discarded, as a further safeguard against cross-contamination of samples. Ground samples were air-dried overnight. Twenty grams of each dry, ground sample were placed into a fresh, pre-cleaned sediment jar for analysis by Zenon Environmental. One sample (site number 13) was duplicated, for purposes of check analysis but given separate identification numbers. Zenon Environmental was not informed of this duplication, and the duplicates were treated as separate samples throughout the entire analytical procedure.

EXTRACTION

20 g of dried soil were accurately weighed and mixed thoroughly with 30 g of Na_2SO_4 . The homogeneous sample was then charged into a previously extracted glass thimble containing clean glass wool. Pre-cleaned glass wool was placed over the sample and the thimble was placed into a soxhlet extraction apparatus. 400 mL of toluene were used to extract the soil for a period of 35 hours with a cycle time of 15 minutes.

The extraction apparatus was cooled, and the toluene solution removed and reduced in volume to 2-3 mL on a rotary evaporator with solvent exchange into isooctane. The extract was taken for clean-up and analysis of PCDD and PCDF.

CLEAN-UP

A multiple chromatographic column technique was used for clean-up of the concentrated extracts.

The first column was a multilayer column carefully filled with the following: 1.0 g of silica (bottom layer), 2.0 g of 33% 1 M sodium hydroxide on silica, 1.0 g of silica, 4.0 g of 44% concentrated sulfuric acid on silica, 2.0 g of silica and 1 cm sodium sulfate. The column was prewashed with 30 mL of hexane which was discarded. The sample extract was transferred to the column followed by three 5 mL hexane rinses of the flask. An additional 30 mL of hexane was then passed through the column.

The total eluate was collected in a 250 mL flask. Isooctane was added to the eluate which was then concentrated to ca 1 mL.

The concentrated eluate from the first column was then chromatographed on 1% H_2O/AL_2O_3 (basic column of dimensions 5 x 1.5 cm ID. The column was prewashed with 30 mL hexane. The concentrated isooctane eluate was introduced into the column followed by three 2 mL hexane rinses of the flask. The column was eluted with 30 mL of 2% methylene chloride/hexane and collected as fraction A.

The receiving flask was charged and the PCDD and PCDF were eluted from the column using 30 mL of 50% methylene chloride in hexane. This fraction was concentrated on a rotary evaporator to about 1 mL and transferred with hexane rinses to a Reactivial. The extract was then blown down just to dryness with dry nitrogen gas and then taken up in 25 μ L isooctane for GC/MS analysis.

Some extracts contained high quantities of sulfur which was eliminated by making the extract up to 1 mL in a centrifuge tube and shaking with activated copper. Sample extracts which were colored following cleanup were applied to a 1 gm 10% $AgNO_3$ on silica gel column (disposable pipet) in isooctane and eluted with a total of 20 mL hexane. The final extract was blown to dryness as before and made up to 25 μ L isooctane.

INSTRUMENTAL CONDITIONS - PCDD/PCDF ANALYSIS

Gas Chromatography

Injection Port	- 270 ⁰
Split	- Closed
Septum Sweep	- 4 mL min ⁻¹
Column Flow	- He @ 20 cm/sec.
Column	- 30 M x .25 mm DB-5

Oven Temperature Profile

80⁰ - 2 min --220⁰ @ 20⁰/min.

220⁰--290⁰ @ 8⁰/min hold 20 min.

GC/MS Interface - Direct Couple

Transfer Area - 270⁰

Mass Spectrometry

Ionization Mode	- Electron Impact
Electron Energy	- 70eV
Filament Emission	- 0.5A
Electron Multiplier	- 1400V @ 5 x 10 ⁶ Gain
Ionizer Temperature	- 100 ⁰
Scan - Stepped Multiple Ion Detection Procedure	

PCDD/DF MID PROCEDURE

AUTOMD	= ACQU (I; & TC: 01650; S: E)
	= WAIT #25
	= BEEP: BEEP
	= WAIT #840
	= ACQU (S: E)
	= WAIT #980
	= ACQU (&P5; E)

= WAIT #1110
 = ACQU (&H6; E)
 = WAIT #1250
 = ACQU (&H7; E)
 = WAIT #1450
 = ACQU (&OD: E)
 = WAIT #1620
 = BEEP; BEEP; BEEP; BEEP; BEEP

CRITERIA FOR IDENTIFICATION

1. Quantitation Ions & Ion Ratio Requirements

Parameter	Ions (m/z)	Ratio (M/M ⁺²)	
TCDD	320,322	0.7	.11
2,3,7,8-TCDD ¹³ C ₁₂	332,334	0.77	.11
TCDF	304,306	0.77	.11
P ₅ CDD	354,356	0.60	.10
P ₅ CDF	338,340	0.60	.10
H ₆ CDD	388,390	0.49	.08
H ₆ CDF	372,374	0.49	.08
H ₇ CDD	424,426	1.05	.15
H ₇ CDF	408,410	1.05	.15
OCDD	458,460	.93	.14
OCDF	442,444	.93	.14
OCDD ¹³ C ₁₂	470,472	-	

2. Retention Time & Response

- i) Signal to noise ratio minimum of 3:1
- ii) Specific isomer analyses - retention time within 1%
- iii) Non specific isomer analysis - retention time no further than 10% scans from available standard ($T_4 - H_6$) and no further than 5% scans from available standards (H_7).

METHOD OF QUANTIFICATION

Quantitation was performed according to external responses as follows:

TCDD	- 2,3,7,8-TCDD
TCDF	- 2,3,7,8-TCDD
P_5 CDD	- 1,2,3,7,8- P_5 CDD
P_5 CDF	- 2,3,4,6,7,8- H_6 CDF
H_6 CDD	- 1,2,3,4,7,8- H_6 CDD
H_6 CDF	- 1,2,3,4,7,8- H_6 CDF
H_7 CDD	- 1,2,3,4,6,7,8- H_7 CDD
H_7 CDF	- OCDF
OCDD	- OCDD
OCDF	- OCDF

All data were corrected for percent recoveries by MOE staff as follows: tetra, penta-, and hexachlorinated congeners were adjusted for the recovery of ^{13}C -2,3,7,8-TCDD, hepta and octachlorinated congeners were corrected for the recovery of ^{13}C -OCDD.

CHLORINATED DIOXINS/FURANS IN SOIL SAMPLES**

(all concentrations in parts-per-trillion)

Lab./Field Nos.	2338	2339	2340	2341	2342	2343	2344	2345	2346	2347	2348	2349	2350*	2350*	2351	2352
Site Nos.	12	8	3	2	11	4	5	9	6	10	1	7	13	13	ash	14
T_4 CDD(0.3) ^a	nd	nd	nd	nd	nd	nd	7	nd	nd	nd	nd	nd	nd	nd	250	nd
P_5 CDD(1.3)	nd	nd	nd	nd	580	nd	nd	nd	nd	nd	nd	nd	nd	nd	160	nd
H_6 CDD(1.3)	nd	nd	nd	nd	170	nd	nd	nd	nd	nd	nd	nd	nd	nd	330	nd
H_7 CDD(1.3)	5	97	270	nd	390	42	42	nd	nd	150	nd	nd	110	82	1100	nd
OCDD(1.3)	940	3200	690	310	3500	1300	140	50	120	nd	75	1000	140	79	920	810
Total Dioxins	950	3300	960	310	4600	1300	190	50	120	150	75	1000	250	160	2800	810
T_4 CDF(0.3)	9	68	nd	nd	nd	nd	nd	nd	43	nd	nd	nd	50	91	850	nd
P_5 CDF(1.3)	6	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	180	nd
H_6 CDF(1.3)	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	140	nd
H_7 CDF(1.3)	nd	nd	150	nd	180	nd	nd	nd	nd	nd	nd	nd	nd	nd	260	nd
OCDF(0.8)	nd	81	nd	nd	nd	5	nd	nd	nd	nd	nd	33	nd	18	1500	nd
Total Furans	15	150	150	nd	180	5	nd	nd	43	nd	nd	33	50	110	2900	nd
Total Dioxins + Furans	960	3400	1100	310	4800	1300	190	50	160	150	75	1000	300	270	5700	810
% Recovery -																
^{13}C -TCDD	141	121	42	42	60	80	179	145	54	98	138	46	112	86	95	107
^{13}C -OCDD	234	148	74	184	88	77	261	314	83	nd	122	75	67	90	49	24

** All values are corrected for recoveries

* duplicate analyses

^a numbers in brackets are average detection limits in parts-per-trillion

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